COAL ASH RESOURCES RESEARCH CONSORTIUM

1993–1998 Final Report

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EXECUTIVE SUMMARY

The Coal Ash Resources Research Consortium (CARRC, pronounced "cars") is the core coal combustion by-product (CCB) research group at the Energy & Environmental Research Center (EERC). CARRC focuses on performing fundamental and applied scientific and engineering research emphasizing the environmentally safe, economical use of CCBs. This consortium of member organizations, scientists, and engineers has a history of ten years of addressing issues related to CCB utilization and disposal. CARRC member organizations, which include utilities and marketers, are key to developing industry-driven research in the area of CCB utilization and ensuring its successful application. The EERC team approach is emphasized in every aspect of the research effort. CARRC continued the partnership of industry partners, university researchers, and the U.S. Department of Energy (DOE) addressing needs in the CCB industry through technical research and development projects. Technology transfer also continued through distribution and presentation of the results of research activities to appropriate audiences, with emphasis on reaching government agency representatives and end users of CCBs. DOE is a partner in CARRC through the EERC Jointly Sponsored Research Program (JSRP) which provides matching funds for industrial member contributions and facilitates an increased level of effort in CARRC.

CARRC partners have evolved technically and have jointly developed an understanding of the layers of social, regulatory, legal, and competition issues that impact the success of CCB utilization as applies to the CCB industry in general and to individual companies. CARRC researchers and members feel confident in their ability to answer technical questions about CCB utilization that may be posed by potential users, regulators, and environmentalists. Technical research on CCBs is successful when it adds to the CCB information pool. Many CARRC tasks are designed to provide information on CCB performance including environmental performance, engineering performance, favorable economics, and improved life cycle of products and projects. CARRC technical research tasks are developed based on member input and prioritization.

CARRC activities from 1993–1998 included a variety of research tasks, with primary work performed in laboratory tasks developed to answer specific questions or evaluate important fundamental properties of CCBs. The tasks summarized in this report are 1) The Demonstration of CCB Use in Small Construction Projects, 2) Application of CCSEM (computer-controlled scanning electron microscopy) for Coal Combustion By-Product Characterization, 3) Development of a Procedure to Determine Heat of Hydration for Coal Combustion By-Products, 4) Investigation of the Behavior of High-Calcium Coal Combustion By-Products, 5) Development of an Environmentally Appropriate Leaching Procedure for Coal Combustion By-Products, 6) Set Time of Fly Ash Concrete, 7) Coal Ash Properties Database (CAPD), 8) Development of a Method for Determination of Radon

Hazard in CCBs, 9) Development of Standards and Specifications, 10) Assessment of Fly Ash Variability, and 11) Development of a CCB Utilization Workshop.

CARRC topical reports were prepared on several completed tasks: “Comparison of State Department of Transportation Specifications for Coal Ash Utilization,” “Sulfate Resistance of Fly Ash Concrete: An Overview of Selected Publications,” and “Scaling Resistance of Portland Cement Concrete Containing High Levels of Coal Combustion Fly Ash.”

CARRC research activities were enhanced by a variety of complementary activities, including CCB research projects funded from several different sources. Several of these projects were performed in an effort to allow members and nonmembers to fund small research projects through CARRC. These projects are jointly funded by the DOE JSRP at the EERC and are selected to provide information valuable to the CARRC research group. CARRC researchers’ participation in activities of the American Coal Ash Association (ACAA), the American Society for Testing and Materials (ASTM), and the Region 8 Ash Utilization Group also enhanced the CARRC research and allowed the results to be used more readily by the CCB industry. CARRC researchers participated in numerous regional, national, and international conferences and symposia as part of the ongoing effort to communicate with CCB producers, marketers, and end users. CARRC participation included presentations and developing and chairing conference series and sessions. CARRC researchers promoted CCB utilization through example by encouraging its use in University projects and even personal construction projects. These projects allowed CARRC researchers to work directly with end users of CCBs and develop a better understanding of their concerns.

In 1993, CARRC researchers also prepared a report for DOE titled “Barriers to the Increased Utilization of Coal Combustion/Desulfurization By-Products by Governmental and Commercial Sectors,” facilitating DOE’s preparation of a Report to Congress on barriers to CCB utilization which was important to the CCB industry. That report was updated in 1998 and submitted for publication by DOE. While the preparation of these reports was a separate effort from CARRC, the CARRC members were instrumental in providing vital information for the two reports.

The primary goal of CARRC is to work with industry to solve CCB-related problems and promote the environmentally safe, technically sound, and economical utilization and disposal of these highly complex materials. Individual goals are set on an annual basis, and CARRC researchers have successfully achieved the majority of annual technical and technology transfer goals with contributions of materials, technical guidance, and information on industry perspective from CARRC members.

CARRC 1993–1998 accomplishments included:

- Updating the CAPD to a user-friendly database management system, and distributing it to CARRC members.

- ASTM standard preparation for a guide to using CCBs as waste stabilization agents.

- Preliminary identification of specific mineral transformations resulting from fly ash hydration.

- Limited determination of the effects of fly ash on the set time of concrete.
- Statistical evaluation of a select set of fly ashes from several regional coal-fired power plants.

- Development and presentation of a workshop on CCB utilization focused on government agency representatives and interested parties with limited CCB utilization experience.

- Participation in a variety of local, national, and international technical meetings, symposia, and conferences by presenting and publishing CCB-related papers.

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1.0 INTRODUCTION

The Coal Ash Resources Research Consortium (CARRC) program has evolved since it began 13 years ago. This evolution is the result of the direction and input of the industrial members and their representatives who participate in a CARRC advisory board. CARRC industry members provide financial support, which is matched by the U.S. Department of Energy (DOE) through the Energy & Environmental Research Center (EERC) Jointly Sponsored Research Program (JSRP). Input from CARRC members also facilitates the development of other EERC coal combustion by-product (CCB)-related research projects.

1.1 CARRC Industrial Members

The 1993–1998 members listed below are gratefully acknowledged for their participation and cooperation in CARRC research tasks and in other EERC efforts:

Brett Admixtures, Inc.
Cooperative Power Association
National Minerals Corporation/Mineral Solutions
Nebraska Ash Company
Northern States Power Company
Otter Tail Power Company

CARRC also acknowledges Dr. Jerry Harness and Ms. Susan Joines, formerly the CARRC DOE Performance Monitors. The current DOE Performance Monitor is Mr. Robert Patton.

1.2 CARRC Research Staff

The EERC's multidisciplinary approach to research is well demonstrated in CARRC research and related activities. CARRC has the opportunity to draw from the diverse research staff at the EERC while maintaining a core staff that focuses on coal by-product utilization and disposal research. Key to this approach is communication among numerous individuals and groups as well as the coordination of sample identification, collection, distribution, and data manipulation. The core EERC CARRC research group consists of the following individuals:

Bruce A. Dockter, Research Engineer
Kurt E. Eylands, Research Associate
David J. Hassett, Senior Research Advisor
Erin M. O'Leary, Information Systems Manager
Debra F. Pflughoeft-Hassett, Research Manager

North Dakota State University (NDSU) CARRC research is performed by Gregory J. McCarthy, Professor of Chemistry and Geology.

The CARRC primary consultant is Oscar E. Manz, Professor Emeritus, Civil Engineering, University of North Dakota, and Manz Associates.

CARRC also employs and trains graduate and undergraduate students at the University of North Dakota (UND), NDSU, and other universities and colleges through the Associated Western Universities (AWU) and other exchange programs. These students were directed by CARRC researchers and provided valuable information and skills that enhanced CARRC activities. A primary advantage of the work performed by these students is the knowledge and experience they take with them as they move into technical and professional careers. CARRC researchers wish to acknowledge the following students for their role in advancing CARRC research during 1993–1998:

Wade J. Heintz, undergraduate, Mechanical Engineering and Engineering Management, UND
Kevin A. Kuhl, graduate student, Civil Engineering, UND
Wayne L. Gerszewski, graduate student, Civil Engineering, UND
Michael J. Morris, undergraduate, Chemical Engineering, UND
Bryan G. Bocht, undergraduate, Civil Engineering, UND
L. Cava, undergraduate, Civil Engineering, UND
Glenn R. Gustafson, undergraduate, Civil Engineering, UND
Bryan Corbitt, undergraduate, Chemistry, AWU, Gilford College, Greensboro, NC
Kari J. Espelien, undergraduate, Information Management, UND
Lih M. Chua, undergraduate, Civil Engineering, UND
Dean Grier, undergraduate, Geosciences, NDSU
Jodi Parks, postgraduate fellow, Geosciences, NDSU
B. Jarabek, undergraduate, Chemistry, NDSU
James C. Alstatt, undergraduate, Mechanical Engineering, UND
Heather E. Holden, undergraduate, UND
Dustin Kouba, Red River High School
Karin Laursen, graduate research scholar, Denmark
Angela L. Hedgecough, undergraduate, Geology, UND

EERC laboratory facilities and their associated technical staff play important roles in performing CARRC research. The EERC laboratories most closely involved in CARRC research are listed below:

Analytical Research Laboratory
Coal Analysis Laboratory
Coal By-Products Utilization Laboratory
Materials Properties Research Laboratory
Natural Materials Analytical Research Laboratory

2.0 BACKGROUND

CARRC continued in its twelfth year of research, development, demonstration, and technology transfer focused on the environmentally safe and technically sound utilization of CCBs. CARRC brings together industry partners, university researchers, and DOE to identify needs in the CCB industry that can be addressed through technical research and development projects and to distribute and present the results of those activities to appropriate audiences, with emphasis on reaching government agency representatives and end users of CCBs. While the initial focus of CARRC was to develop a database of information on coal fly ash, the perspective of members and researchers changed when the Coal Ash Properties Database (CAPD) became a research tool and no longer the primary research effort. The success in developing the CAPD through the CARRC team effort provided impetus to expand the activities performed to include member-driven research tasks that required practical input from members and the technical expertise of CARRC researchers using the available university facilities. CARRC membership has remained relatively constant, with industrial members primarily concerned with the marketing and utilization of moderate- to high-calcium fly ash. These members have facilitated the development of significant information on fly ash chemistry, especially as it relates to the mobility of fly ash constituents on exposure to water, the hydration reactions of moderate- to high-calcium fly ash to improve the understanding of the performance of fly ash in utilization applications, comparative performance of a range of fly ash samples in numerous construction and engineering applications, and the best methods of testing and analyzing CCBs for accurate and reproducible results that are scientifically valid and legally defensible.

In recent years, an emphasis on information transfer has been added to the CARRC priorities in response to member and industry input. It was evident to CARRC members and researchers that technical reports alone did not meet the need for information dissemination. CARRC researchers needed to present results of CARRC activities in a variety of formats and levels of detail. Many current CARRC technology transfer activities are informal and include providing documents and/or verbal comments to government agencies, end users, citizen groups, students, and any other interested parties. The EERC Internet site has encouraged many interactions about CARRC activities and general information on CCBs.

In order to perform research pertinent to CARRC members, they frequently submit samples from their own facilities for inclusion in research tasks. Results of any CARRC tasks are presented in a manner to maintain the confidentiality of CARRC members' samples. Each member receives the experimental results based on their submitted materials with appropriate identification, and this courtesy is also offered to nonmembers who contribute samples for any CARRC research. CARRC members may distribute or use any CARRC information as they wish. One example of data confidentiality and distribution is the CAPD, which contains identification and characterization information on more than 800 CCB samples from U.S. and international sources. The majority of the CAPD entries represent member-submitted materials. CARRC members agreed that certain identification fields should only be accessed by the member who submitted the sample. CARRC provides the CAPD to members on computer disks, and each company has its own version of the CAPD that indicates confidential identifying information for that company's samples only. All other fields are available to all users. The CAPD software allows each company to add data to its own CAPD version. Further, each member may distribute copies of their version of the CAPD as desired.

CARRC members and researchers have progressed together technically but have also jointly developed an understanding of the layers of social, regulatory, legal, and competition issues that impact the success of CCB utilization in a generic sense and with regard to specific projects and applications of significance to a single CARRC member. CARRC researchers and members feel confident in their ability to answer technical questions about CCB utilization that may be posed by potential users, regulators, and environmentalists. Technical research on CCBs is truly successful if it facilitates CCB utilization that meets the performance criteria of the customer. These criteria can include environmental performance, engineering performance, favorable economics, and improved life cycle of products and projects. Members bring to CARRC questions from their daily interactions with customers, end users, and the public. Technical research tasks are developed to address these questions, and members select annual activities by a prioritization ballot. This process limits the number of research tasks annually, but the selected tasks are of highest member priority or need. As stated earlier, CARRC researchers work to make results of technical tasks available to the group or individual who originated the question about CCB utilization.

DOE is a partner in CARRC through the EERC JSRP, which provides matching funds for industrial contributions and increases the level of effort in CARRC. In addition to providing funding, a DOE representative is invited to provide input to technical tasks and other activities. This input is valuable in providing a broad perspective from a federal agency. CARRC DOE representatives have also indicated that interaction with industry representatives provides perspective that is helpful relative to other DOE project areas and interests. DOE participation has been positive for CARRC and for the CCB industry in general.

CARRC is successful because it provides high-quality, focused research with highly leveraged funds relative to individual industrial contributions. CARRC takes advantage of the diverse experience, knowledge, and skills in its membership and research team and provides a flexible approach to research. CARRC continues to evolve to meet the challenges and needs of its members through high-quality technical research and technology transfer.

3.0 OVERALL CARRC RESEARCH OBJECTIVES

The primary objective of CARRC is to work with industry to solve CCB-related problems and promote the environmentally safe, technically sound, and economical utilization and disposal of these highly complex materials. Goals include the generation of scientific and engineering information regarding regulations and specifications for CCBs, the development of improved characterization methods for CCBs, the demonstration of new or improved CCB use applications, and technology transfer.

4.0 ACCOMPLISHMENTS

4.1 July 1, 1993 – June 30, 1995, Tasks

Goals for 1993–1995 were to:

- Advance predictive capabilities for ash behavior in utilization applications.
- Promote high-volume use applications for coal ash.
- Evaluate the potential for environmental impact from coal ash utilization and disposal.
- Develop a database of utilization application requirements.
- Investigate advanced technological utilization applications, and promote innovative ash use.
- Facilitate the production of consistently high-quality coal ash.

The following tasks were undertaken to meet these goals.

4.1.1 Demonstration of Coal Combustion By-Product Use in Small Construction Projects

A significant factor for advancing coal ash use is promoting this by-product in field applications. The intent of the demonstrated use of coal ash through CARRC is to establish a long- term commitment to ash use from commercial and public entities. With this objective in mind, the first step was to establish a standard practice for the UND physical plant services department to use coal ash in all concrete applications on campus. The next step was to promote concrete containing fly ash for construction activities in the city and, eventually, statewide.

Beginning in 1993, the EERC, through efforts supported by CARRC, was able to rewrite the materials specifications for concrete use on the UND campus. The university is the only campus in the state that performs its own engineering design work for in-house construction. All concrete placed since 1993 in sidewalks, parking lots, and pavements has utilized 15% to 70% fly ash as a partial cement replacement. In an annual statewide university plant services meeting held in Grand Forks in 1994, CARRC representatives presented these results to several university representatives from around the state. The reception of these efforts was very positive, and suggestions for coal ash utilization were brought back to represented departments.

In the past 3 years, over 100 concrete cylinders containing fly ash have been tested in the laboratory for compressive strength. The 7-day strengths on these cylinders have varied from

2500 to over 4000 pounds per square inch (psi), while the 28-day strengths have ranged from 3300 to over 5000 psi.

In addition to compressive strength testing, concrete samples were also taken for evaluation for rapid freezing and thawing action. Concrete samples measured approximately 3 in.^{2} × 14 in. long. The freeze–thaw cabinet located in the Coal By-Products Utilization Laboratory at the EERC was fabricated by EERC technicians. The testing apparatus is one of only two located in the state where this type of accelerated freeze–thaw testing can be performed. The other is at the North Dakota Department of Transportation in Bismarck, North Dakota.

All concrete beams for freeze–thaw testing were stored and measured according to Procedure A of American Society for Testing and Materials (ASTM) Standard Test Method C666, Resistance of Concrete to Rapid Freezing and Thawing. The beams were so arranged that except for necessary supports, each specimen was completely surrounded by water at all times while being subjected to freezing and thawing cycles. Measurements taken included length (in.), weight (lb), and transverse frequency (Hz). Calculations performed included percent change in length and weight, relative dynamic modulus, and Young's modulus of elasticity.

4.1.2 Application of CCSEM for CCB Characterization

Coal combustion ashes can be either pozzolanic or cementitious. Pozzolanic materials form compounds with cementitious properties in the presence of CaO (lime) or some other usable source of calcium and water. Cementitious materials self-harden when they come into contact with water. While two coal combustion fly ashes may have similar chemistries, their behavior can be significantly different, with one of the ashes showing pozzolanic properties and the other self-hardening in a matter of a few minutes. These properties can be related to mineralogical and chemical differences. This task was designed to examine the particle chemistry in detail through the use of computer-controlled scanning electron microscopy (CCSEM).

CCSEM is a process that locates a particle that has been mounted in epoxy, measures eight diameters, determines a shape factor, takes a chemical analysis of the center of the particle, and classifies the particles according to its chemistry.

The technique analyzes more than 2000 points per sample, allowing for a statistically valid number of analyses per sample to ensure a high degree of confidence. The raw data collected on the point analyses from the CCSEM process were subjected to cluster analysis, a statistical process that groups the analytical results into groups of similarity based on particle chemistry. For the samples evaluated, 30 significant clusters were identified. Mineral names were not assigned to the clusters—they were named according to the most abundant to the least abundant element present and are meant to show chemical associations among the elements analyzed. The chemistries reflect the mineral phases and the glassy matrix in which they are contained. These data must be used in conjunction with x-ray diffraction (XRD) results to better understand the relationship between the mineral phases and the glass in which they are included.

The identification of various calcium and aluminum mineral phases in cementitious fly ashes significantly increases the ability to predict how these materials will behave once they come into contact with water, whether in a utilization or disposal scenario. The amounts of reactive calcium and aluminum can be better quantified to help predict such properties as resistance to sulfate attack or to determine how much of the mineral ettringite will form. These identifications can also lead to a more meaningful classification of fly ashes based on their cementitious or pozzolanic properties.

4.1.3 Development of a Procedure to Determine Heat of Hydration for CCBs

CCBs are complex, exhibiting characteristics highly advantageous for use in construction and manufacturing applications. The most common application is as a mineral admixture in concrete. The current ASTM classification system for this application does not provide adequate information to assess the reactivity of these materials outside of their use in cement and concrete products. The pozzolanic or cementitious nature of fly ash is normally determined by empirical physical test procedures. The current classification system used for fly ash does not have any indication of pozzolanic or cementitious properties associated with it, although these properties are often implied. Heat of hydration measurement has been used in the cement and coal ash industry for a number of years to predict heat buildup in large-scale construction and to estimate reactivity. The EERC is developing a method using heat of hydration that may be a means to predict the reactivity of coal ash. This method will also be used in conjunction with XRD to aid in understanding the short-term reactions involved in the hydration of fly ash. A continuous-scale rating for pozzolanic/cementitious behavior would provide more adequate information to assess the reactivity of these materials and would facilitate their use in applications beyond cement and concrete products. A test procedure to provide a continuous-scale rating from 0 to 10 for pozzolanic and cementitious behavior based on heat of hydration was proposed for development in this task.

An apparatus was assembled to determine the heat of hydration of CCBs. The technique employs a converted jacketed calorimeter to minimize heat uptake or loss. Temperature measurements were made using a thermistor probe with a digital readout. Experiments were carried out in fleakers or tall beakers stirred with a paddle-type stirrer. Stirring was carried out at a rate that just prevented settling of the ash and was found to contribute minimally to the heat of the system over the time periods studied. To determine the heat of hydration, 100 g of ash was added to 200 g of water. The mass of ash was varied as necessary to provide a nominal temperature rise of 3 to 5 Celsius degrees during the course of the experiment. The thermistor used for temperature readout and a sensing probe that controlled the jacket temperature of the calorimeter were placed into the water prior to the addition of the ash to establish a baseline temperature and equilibrate the jacket. After the addition of ash, the open top of the water-jacketed section of the calorimeter was covered to minimize heat gain or loss. The jacket and control assembly were able to maintain the temperature of the jacket to within several thousandths of a Celsius degree of the temperature of the hydrating ash. Drift of the system using only stirred water was minimal, at less than 0.001 Celsius degree per minute, since heat input from stirring was taken into account by carefully controlling the jacket temperature. Before each experiment, the ash and water were allowed to equilibrate to the same temperature. Readings of temperature were taken immediately and at intervals of 1 minute. Experiments were terminated when the heat evolution had decreased to a steady but minimal rise during each time period. A nominal cutoff point of 0.002 to 0.003 Celsius degrees of increase per minute was chosen as the rate at which to terminate the experiments. Most experiments were carried out for between 1 and 3 hours. Duplicate experiments were performed on five archived coal fly ash samples from participating CARRC members. Results of these experiments were evaluated and compared with other existing characterization results on those samples.

Heat of hydration curves and first-derivative plots were developed. Two major trends were noted in the samples evaluated. The first shows a spike at the beginning of the reaction of ash with water that decays to what would be referred to as a nominal slow rise in temperature associated with a slow decay. At the point where the heat rise stabilizes, with a slight decrease with respect to time, the experiments were terminated. A second trend shows this initial spike followed by several other rate increases, ending with the gradual slow decrease, at which time the experiments were terminated.

The use of heat of hydration measurement along with mineralogical characterization as well as cluster analysis of CCSEM results is a first step in expanding our understanding of fly ash hydration reactions. Continued work in this task will be performed in conjunction with the task on CCSEM. Using inflection points in the first-derivative curves, it will be possible to perform XRD characterizations at specific time intervals and collect information, which is the first step in understanding ash hydration.

4.1.4 Investigation of the Behavior of High-Calcium CCBs

As a class of materials, high-calcium CCBs (e.g., Class C fly ash, flue gas desulfurization by-products, and by-products from fluidized-bed combustion) normally exhibit cementitious properties. The principal cement binder in high-calcium CCB cementitious systems is ettringite, $Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$. The binder in these materials is being referred to as "sulfopozzolanic" to contrast it with the "silicopozzolanic" binder in conventional cements. Utilization of these materials depends largely on these cementitious properties.

The objectives of this task during the 1993–95 period were to 1) complete a screening study of the hydration behavior, physical properties, and potential for hazardous element solidification and stabilization of selected CCBs and 2) initiate studies of the behavior of CCBs in atmospheres and solutions containing normal and elevated levels of carbon dioxide and sulfate. The overall goal of this task is to develop a predictive model for the behavior of CCBs in disposal and civil engineering utilization environments.

Work addressing the first objective, the screening study of the hydration behavior, physical properties, and potential for hazardous element solidification and stabilization of selected CCBs, was completed. The studies are described in two publications as follows:

- McCarthy, G.J.; Solem-Tishmack, J.K. "Hydration Mineralogy of Cementitious Coal Combustion By-Products," in *Advances in Cement and Concrete*; Grutzeck, M.W.; Sarkar, S.L., Eds.; American Society of Civil Engineers: New York, 1994; pp 103–122.

- Solem-Tishmack, J.K.; McCarthy, G.J.; Dockter, B.A.; Eylands, K.E.; Thompson, J.S.; Hassett, D.J. "High-Calcium Coal Combustion By-Products: Engineering Properties, Ettringite Formation, and Potential Application in Solidification and Stabilization of Selenium and Boron," *Cement Concrete Res*. **1995**, *25*, 658–670.

Concurrent with these studies, multiyear field tests and core characterization performed by EERC–NDSU personnel, collaborating with Radian Corporation on a DOE project, have shown that several types of high-calcium sulfopozzolanic materials have significant loss of strength and increase in permeability, accompanied by major changes in hydration mineralogy.

Work addressing the second objective, study of the behavior of CCBs in atmospheres and solutions containing normal and elevated levels of carbon dioxide and sulfate, was initiated in the third quarter of 1994. The materials tested had all been prepared during previous projects. They were hydrated CCBs or CCBs that had been recovered as cores from the field test project noted above.

These materials were exposed to ambient levels of CO_2 (i.e., 0.035% CO_2 in air) and a controlled atmosphere of 7.5%CO_2–92.5%N_2. Three relative humidity (RH) conditions were used: <5% RH controlled by a desiccant in the exposure chamber; >95% RH controlled by free water in the chamber; and ambient (uncontrolled) laboratory RH. All tests were carried out at laboratory ambient temperatures (20°–27°C over the year). Ettringite and other crystalline phases were monitored by powder XRD.

The results of this study can be summarized as follows.

<u>In 7.5% CO_2–92.5% N_2 atmosphere with >95% RH</u>

- We have confirmed that the following "acid–base neutralization" reaction of ettringite and the ettringite binder in CCBs occurs in a matter of hours to days at >95% RH:

$$\underset{\text{Ettringite}}{Ca_6Al_2(SO_4)_3(OH)12 \cdot 26H_2O} + 3CO_2 \rightarrow \underset{\text{Calcite, Aragonite, or Vaterite}}{3CaCO_3} + \underset{\text{Gypsum}}{3CaSO_4 \cdot 2H_2O}$$

$$+ \underset{\text{Amorphous}}{2Al(OH)_3} + 23H_2O$$

- Synthesized ettringite decomposes completely within 48 hours.

- Ettringite in hydrated CCBs and previously landfilled core CCBs initially exhibited rapid decomposition (at least 50% by weight) within the first 96 hours, with complete degradation occurring within 3 to 161 days, depending on the sample. The North Dakota high-sodium sample is a notable exception, having retained residual ettringite for 263 days to date.

- Thaumasite was more resistant to CO_2-induced degradation than ettringite in atmospheric fluidized-bed combustion (AFBC) and limestone injection modified burner (LIMB) core samples. However, the onset of thaumasite decomposition was observed at 33 and 95 days, respectively.

- Gypsum and calcium carbonate formation was observed in association with the loss of ettringite (and thaumasite, where present).

- In general, calcite was the most common form of calcium carbonate produced. However, aragonite appeared as the dominant polymorph in the synthetic ettringite, as well as both the high- and low-sodium North Dakota samples. Lower concentrations of calcite and vaterite were also observed in these three instances, with no noticeable increases throughout the study.

- Portlandite, as expected, reacted completely to form calcite within the first 3 days in AFBC samples.

In ambient levels of CO_2 and >95% RH

- Synthesized ettringite showed only minimal degradation during the first 56 days, resulting in the formation of minor calcite, aragonite, and gypsum. By Day 104, ettringite had decreased to approximately 4 wt% of the crystalline content. The other phases present were gypsum and nearly equal amounts of calcite, aragonite, and vaterite.

- Ettringite in core samples of CCBs was observed to undergo significant decomposition beginning after 20–40 days of exposure.

- Thaumasite remained stable in the AFBC and LIMB CCBs to 159 and 165 days, respectively.

- Gypsum and calcium carbonate formation was observed in association with the loss of ettringite (and thaumasite, where present).

- In general, calcite was the most common form of calcium carbonate produced. Low concentrations of aragonite and vaterite were also observed in the lignite ash samples, with no noticeable increases throughout the study.

- In the AFBC samples, significant carbonation of portlandite to calcite occurred within the first 40 days. Interestingly, the onset of significant loss of ettringite occurred after this carbonation of portlandite was complete.

4.1.5 Development of an Environmentally Appropriate Leaching Procedure for CCBs

The synthetic groundwater leaching procedure (SGLP) was developed as a generic leaching test to be applied to materials to simulate actual field leaching conditions for either disposal or utilization situations. Since the toxicity characteristic leaching procedure (TCLP) was designed to simulate leaching in a sanitary landfill under codisposal conditions, it is not appropriate to evaluate leaching of CCBs in typical disposal or utilization scenarios. To provide more appropriate and predictive information for CCBs and other unique materials, a leaching test was developed using the same basic protocol as the TCLP, but allowing for the appropriate leaching solution chemistry. Test conditions are end-over-end agitation, a 20-to-1 liquid-to-solid ratio, and an 18-hour equilibration time. Local, site-specific factors, including geochemical factors likely to influence trace element mobility, would have to be considered for specific cases. Frequently, the most likely source of water would be rainwater; thus prior mineralization would not be a consideration.

A long-term leaching procedure (LTL) has also been developed and used in CARRC and other research projects. The LTL uses the same test conditions as the SGLP, but separate samples are analyzed after longer leaching times, generally 1 and 3 months in duration, since certain coal conversion solid waste materials form secondary hydrated phases with mineralogical and chemical compositions different from any of the material in the original ash.

In preparation for formalizing the SGLP/LTL procedure, a preliminary draft of the SGLP/LTL procedure has been prepared in ASTM format. Review of this document has been initiated, and discussions with the American Coal Ash Association (ACAA) task force on ASTM procedures were held. A list of individuals interested in assisting with the laboratory verification of the procedure was also developed.

4.1.6 Set Time of Fly Ash Concrete

The evaluation of the set time of fly ash concrete was initiated in 1993. The intent of this project was to examine the effects fly ash and chemical admixtures have on the set time of portland cement concrete. Different sources and quantities of fly ash, chemical admixture, and cement were examined.

There are four ASTM test procedures predominantly used for determining set time of cementitious mixtures.

The materials used consisted of seven types of chemical admixtures, five sources of cement, and three sources of fly ash. The chemical admixtures were combinations of water-reducing agents, set retardants, nonchloride accelerators, and mid-range water reducers. With this many variables being examined, there would be over 100 different mixture combinations to evaluate. To reduce the potential number of mixtures, combinations of cement and fly ash considered most probable to be combined in the field were evaluated first using the chemical admixtures.

To establish a baseline time-of-set for each trial mixture, all five cements and cement and fly ash combinations were evaluated to establish a “control” final setting time. The control setting time results were the reference times used in evaluating the various chemical admixtures with cement and cement–fly ash combinations. The normal consistency of each paste mixture was obtained according to ASTM C187, Test Method for Normal Consistency of Hydraulic Cement. All mixtures containing cement and fly ash had 20% by weight of the cement replaced with fly ash.

General trends were obvious in evaluating the cement and fly ash mixtures without the addition of any chemical admixtures. Less water was required to obtain the normal consistency when a combination cement and fly ash mixture was used. All mixtures using fly ash required more time to reach a final set than for cement pastes using no fly ash. These trends are what would normally be expected.

However, no significant trends could be seen in incorporating the chemical admixtures. All chemical admixtures, with the exception of one, required similar quantities of solution to obtain the required setting times. One of two nonchloride accelerator agents did require significant additional amounts of solution. This accelerator agent should have actually resulted in one of the shortest setting times. The longer time may be due to such small quantities of chemical admixture being used in each mixture. These additions were based on the quantities of dry materials used in each mixture. Because of these unrealistic results, the testing program was discontinued until further review of the task structure could be conducted.

A better method of investigation is needed to compare laboratory results to concrete characteristics experienced in the field. The first recommendation is to use only one chemical admixture, such as one of the nonchloride accelerators. The intent is to reduce the total number of mixtures to evaluate. A second recommendation is to use a different method of determining the time of set. To more closely correlate laboratory results to those expected in the field, ASTM Test Procedure C403 should be used. This method requires the preparation of a concrete mixture instead of a paste. Larger quantities of materials are needed, so this is an additional reason for reducing the number of total mix designs.

One characteristic of the ASTM C403 procedure is that excess water in the concrete matrix is allowed to accumulate at the surface of the fresh, curing concrete. This is a natural occurrence in concrete placement and is commonly referred to as “bleed water.” The procedure calls for the bleed water to be removed and measured during the curing process. This is an important element in the investigation of concrete curing and setting.

4.1.7 Coal Ash Properties Database

The CAPD was originally created as a spreadsheet program called the Western Fly Ash Database. The database consisted of physical, chemical, and mineralogical results from the analysis of samples submitted to the EERC from CARRC members. A limited amount of operational and materials data were also entered in the database for each sample. The database was primarily used by researchers in developing an understanding of coal ash variability and behavior. The database allowed EERC researchers to sort and graph the analytical data.

The number of samples and the amount of identifying information for each sample quickly increased, and samples were no longer limited to western coal fly ash, but included fly ash and other CCBs from both U.S. and international sources. In addition, CARRC members were interested in an efficient method to directly access the data at their locations. The spreadsheet format was limiting and not suitable for complex data queries. Therefore, the spreadsheet program was replaced by a relational database program and renamed the “Coal Ash Properties Database.”

CAPD was designed using a graphical user interface, providing user-friendly point-and-click interaction with the program. Complex queries can be generated through a series of pop-up menus and dialog boxes. Once the query has been defined, the data can be viewed in a variety of formats, including comparison of data to ASTM C618 references and a column display of specific data fields for correlation of analytical results. The database contains several standard reporting options. All selected data can be directed as output to an ASCII file for use in other computer programs. The first version of CAPD containing over 700 samples was distributed to CARRC members at the annual meeting in May of 1993.

The focus of activities over the past 2 years was on expanding the descriptive and analytical information collected for each sample, increasing the number of samples, and improving the user interface.

The database was expanded to include more descriptive sample information fields. Identifying information about the sample and the conditions under which the sample was formed is essential for researchers who use the analytical data to make correlations between fuel properties, operating conditions, and CCB characteristics and performance. The comprehensive characterization of CCBs provides valuable information relevant to the use of the materials in numerous applications, including those in the engineering and construction areas, and the potential for environmental impact upon utilization.

A collection form was created for the efficient collection and entry of new samples. The form was submitted to CARRC members. Members were encouraged to provide information on samples that were analyzed outside of the EERC for the database. Over 100 new samples were analyzed at the EERC for bulk chemistry, mineralogy, and physical and engineering properties and added to the database. CAPD now contains analytical data on over 800 samples.

The database interface was improved in several ways. The run-time engine was upgraded to the most current version provided by Microrim, Inc. New capabilities were added so that users can input sample data at their locations and can edit and delete existing sample data. An installation program was developed, and the user manual was revised. CAPD Version 3.0 was distributed to users at the annual meeting in 1994.

A select number of samples were analyzed using advanced mineral and characterization techniques. These data were added to a demonstration version of the database for presentation at meetings and conferences.

4.1.8 Technology Transfer

CARRC topical reports were prepared on several completed tasks:

- "Comparison of State Department of Transportation Specifications for Coal Ash Utilization"
- "Sulfate Resistance of Fly Ash Concrete: An Overview of Selected Publications"
- "Scaling Resistance of Portland Cement Concrete Containing High Levels of Coal Combustion Fly Ash"

CARRC researchers worked with other EERC and UND personnel, CARRC member representatives, representatives of state and federal agencies, and other industry groups and representatives in ash-related efforts throughout the year. Key activities included the following:

- CARRC researchers worked with other EERC personnel to provide information to DOE for a report to Congress on the barriers to the increased utilization of coal combustion and desulfurization by-products by government and commercial sectors.

- CARRC researchers participated in the ASTM E50 committee on Environmental Assessment. CARRC participation focused on the E50.03 subcommittee's work on developing a standard guide for use of coal ash in structural fill and in initiating standards for the use of CCBs in controlled low-strength material and in waste solidification/ stabilization. CARRC extended its ASTM voting membership to include D34 and several D34 subcommittees working in areas related to waste management and CCB utilization and disposal. CARRC Consultant Oscar Manz participated on ASTM Committees C1 on Cement, C7 on Lime, C9 on Concrete and Concrete Aggregates, D18 on Soil and Rock, and D34 on Waste Management. Mr. Manz keeps CARRC researchers abreast of the actions of these committees.

- CARRC maintained its associate membership in ACAA and continued to participate in quarterly workshops and committee meetings.

- CARRC researchers worked with UND to change concrete specifications to include coal fly ash. Fly ash utilization projects continued on the UND campus. A CARRC researcher used 70% fly ash concrete in a small construction project for his home. While this was not funded by CARRC, it allowed CARRC researchers to promote high-volume fly ash concrete to contractors and other homeowners.

- CARRC researchers continued participation in the Region 8 Coal Ash Utilization Group.

- The EERC completed a white paper study on the use of coal ash in waste solidification and stabilization under contract to ACAA.

- CARRC research staff worked with the Minnesota Pollution Control Agency Task Force and a coalition of Minnesota utilities to develop recommendations for CCB utilization rules in Minnesota.

CARRC also supported work presented in two Master of Science theses, and twelve papers were published and/or presented by CARRC researchers during 1993–1995.

4.2 July 1, 1995 – December 31, 1996, Tasks

1995–1996 CARRC activities will focus on increasing ash utilization options and facilitating the production of consistent, quality coal ash. Goals for 1995–1996 were to:

- Advance predictive capabilities for and demonstrate ash behavior in utilization applications.

- Develop valid environmental test procedures for coal ash applicable to utilization and disposal.

- Promote product performance to evaluate products containing coal ash.

- Investigate advanced technological utilization applications, and promote innovative ash use.

4.2.1 Demonstration-Scale and Commercial Coal Ash Utilization Projects

CARRC researchers have traditionally worked with regional ash users to encourage the expanded use of conventional coal ash applications, primarily concrete and concrete products. During 1995 and 1996, several ash utilization projects were completed to assess the optimal level of fly ash that could be used in the construction of sidewalks, parking lots, curbs and gutters, and dumpster pads on campus at UND. Most experimental mix designs contained 30%, 50%, and 70% fly ash replacement of cement in concrete. The current specifications for using fly ash in concrete applications at UND are 30% fly ash replacement of cement and all concrete must contain fiber mesh reinforcement. The minimum required 28-day compressive strength of the concrete is 3500 psi, with results generally being in the range of 4000–4500 psi. The information gained in this work has been presented to regional organizations including the Department of Transportation, local engineering consultant firms, and other North Dakota universities for their own local construction activities. Demonstration-scale related work was suspended for 1997, but CARRC researchers will continue to encourage CCB utilization through promotional activities and individual contacts. CARRC researchers frequently respond to requests for general information on CCBs and use applications, CCBs with specific characteristics, standard testing, and other information. CARRC researchers provide public domain documentation, contacts, and CARRC research results when approved by CARRC members.

4.2.2 Quantitation of Coal Ash Reactivity

The following two activities progressed as parallel methods development during 1995–1996, and it was concluded that these techniques used jointly are expected to provide valuable information on hydration reactions in CCBs. This work will proceed in 1997 because CARRC members rated the proposed task high in the annual prioritization process.

4.2.2.1 Application of CCSEM for Coal Ash Characterization

CCSEM is an excellent analytical tool for characterizing coal combustion ashes when used in conjunction with XRD. The CCSEM process allows a large number (>2500) of individual fly ash spheres to be sized, shaped, and classified according to its chemistry. Chemical information on the amorphous (nonstructured) portions of the individual spheres can be related to particle size, and when used in conjunction with XRD, information on conditions of mineral formation can be inferred. CCSEM results can be clustered into like groups based on composition and correlated to the results of XRD.

On hydration, fly ash experiences mineralogical transformations. The minerals quartz, merwinite, and periclase remain unchanged, but calcite and ettringite appear, and anhydrite, mullite, and spinel are absent. CCSEM data have been used to show the rearrangement of the elements. SO_3, K_2O, Fe_2O_3, and CaO are more concentrated in the hydrated fly ash, and calcite and ettringite are represented in the CCSEM data.

The behavior of fly ash in engineering applications is a direct result of the mineral content and hydration reactions. As these reactions take place, energy is often released in the form of heat. The rate at which heat is generated is directly related to the rate at which these mineral transformations take place. CCSEM analyses can be used to evaluate which mineral components are being formed in the various glassy spheres that make up fly ash. CARRC researchers have begun to correlate the information from CCSEM and XRD with the heat of hydration. Further work is planned to combine this task and the following task on heat of hydration. A more useful classification system for coal combustion fly ash based on fly ash reactivity will result.

4.2.2.2 Development of Heat of Hydration Procedure

Heat of hydration has been used in the cement and CCB industries to predict buildup of heat in large-scale use applications, but this task was initiated because CARRC researchers proposed that the heat of hydration information could be more useful in understanding the behavior of fly ash in various applications. Methodology was developed to precisely measure the heat of hydration of fly ash (to the nearest 0.001°C), and samples submitted by CARRC members were evaluated. Temperature measurements were typically recorded at 1-minute intervals for 1–3 hours. It was observed that fly ash samples had heat of hydration “fingerprints.” The heat of hydration behavior was interpreted using first-derivative plots of the data. Derivative curves indicated the rate of heat of hydration. Several trends were observed in the derivative plots and were noted in these evaluations.

The heat of hydration results were interpreted in conjunction with information from CCSEM and XRD. Events noted on the first-derivative plots were correlated to hydration reactions such as the hydration of mayenite, a specific calcium aluminate mineral, and ettringite formation. Hydration reactions are important to the behavior of fly ash in many common engineering applications.

Continued work will determine if heat of hydration can be effectively interpreted with CCSEM and XRD to identify and further characterize hydration reactions. A classification system for coal combustion fly ash partially based on heat of hydration results will be proposed.

4.2.3 Development of Environmentally Appropriate Leaching Procedure for Coal Ash

A draft standard method for the SGLP and optional LTL procedure was prepared in 1993–1995. During the 1995–1996 reporting period, the standard was introduced for consideration as a standard for ASTM E50. The consensus of the appropriate subcommittee, E50.03, was that leaching procedures are not consistent with the goals and focus of the subcommittee, so the draft standard was withdrawn from consideration.

In response to an Environmental Protection Agency (EPA) solicitation, CARRC researchers proposed further development and validation of the procedure, but that proposal was not selected in EPA's competitive process. CARRC researchers regularly receive requests for the SGLP method and for guidance in interpreting results of the method, so it was determined that a CARRC topical report will be prepared and distributed.

4.2.4 Development of a Method for Determining Radon Hazard in CCBs

National and regional concern by citizen groups and regulatory agencies regarding the radionuclide content of CCBs and the potential radon hazard prompted a limited effort through CARRC to develop a reliable method to determine radon hazard in CCBs.

Preliminary discussions with CARRC members and other industry contacts determined that this method development would not be the best approach for CARRC members. Experts in the radionuclide content of coal and CCBs were contacted and agreed that the radionuclide content of CCBs is generally very low and the radon hazard similarly is comparable to many natural materials. It was proposed by these individuals that the sporadic concern regarding these issues was a result of erroneous publications in current literature. CARRC researchers obtained documentation refuting claims of high radionuclide content in CCBs and presented this information to CARRC members at the 1996 CARRC Annual Meeting. It is important to note that variable and sometimes conflicting information on radionuclides in CCBs and coal-fired utility emissions is reported in the reports of DOE’s study of air toxics at utilities across the United States. CARRC researchers and other EERC researchers interested in emission issues plan to perform a critical review of available information. CARRC members will be kept informed of progress as the effort proceeds.

4.2.5 Activities Related to Regulations and Government Agencies

CARRC researchers had the opportunity to interject technical information for consideration in regulatory processes in several states during 1995–1996. While many of these specific efforts were not supported by CARRC funds, research results from past and current CARRC activities provided much of the technical information presented. Since the regulatory process is frequently cumbersome, the direct impact of the CARRC effort in this area is difficult to assess. However, the ongoing support of CARRC members has allowed the generation of scientifically valid and relevant technical information regarding CCBs and their utilization. The result is that CARRC has developed a reputation for providing authoritative information in a concise and easily understood manner for diverse audiences.

In October 1996, CARRC researchers were awarded a contract with DOE to collect and compile technical information on clean coal technology (CCT) by-product characteristics and management practices. The information collected will facilitate industry efforts to provide a comprehensive report to EPA on fluidized-bed combustion by-products. EPA is scheduled to make a final decision on the regulatory status of these materials under RCRA (Resource Conservation and Recovery Act) in 1998. The effort, funded entirely by DOE, will accomplish several important objectives of benefit to CARRC members:

- A database of CCT by-product characteristics will facilitate EPA’s regulatory decision and help potential users and specifiers become familiar with ranges and variability of characteristics of these materials.

- Workshops targeted toward state regulatory agencies will provide a good forum for technology transfer and will encourage dialogue between federal and state agencies and industry.

- The final report summarizing barriers to CCB use will be valuable to the CCB industry, regulators, and CCB users.

4.2.6 Activities Related to Standards Development

CARRC participated in activities of ASTM and the American Concrete Institute (ACI) directly and indirectly in 1995–1996. CARRC researchers focused their efforts on the development of standards that will promote the use of these materials based on advantageous properties. CARRC researchers also work through the required processes to change or eliminate standards that are technically flawed or restrictive to CCB utilization. To accomplish this, CARRC participates in ASTM D34 and ASTM E50 as an organizational member with voting privileges. CARRC input to other ASTM and ACI committees is accomplished through CARRC consultant Oscar Manz and CARRC members.

4.2.7 Technology Transfer

CARRC developed and presented a CCB Utilization Workshop in Bismarck, North Dakota, October 10–11, 1996, in response to member recommendations that educational activities be made a higher priority for CARRC. CARRC researchers were active in ACAA, ASTM, and Western Region Ash Group (WRAG) organization and committee activities. CARRC researchers also gave presentations for primary and secondary school students and for the UND "Family Weekend." Ten papers were published and/or presented by CARRC researchers during 1995–1996.

4.3 January 1, 1997 – December 31, 1997, Tasks

1997 CARRC activities focused on increasing ash utilization options and facilitating the production of consistent, quality coal ash. Goals for 1997 were to:

- Advance predictive capabilities for and demonstrate ash behavior in utilization applications.
- Develop scientifically and legally valid test procedures for coal ash applicable to utilization and disposal.
- Promote beneficial reuse of CCBs through technology and information transfer to industrial, public, and governmental sectors.
- Facilitate development of reasonable CCB standards and regulations, emphasizing product performance and reasonable environmental considerations.

4.3.1 Development of Standards and Specifications

An ASTM Standard Guide for "Use of Coal Combustion Byproducts for Stabilization of Wastes Containing Arsenic, Boron, Chromium, Molybdenum, Selenium, Vanadium, and Other Oxyanionic Species" was prepared and submitted for ballot through the ASTM E50.03 Subcommittee on Global Sustainability/Pollution Prevention. The standard was developed to suggest methods for selection and application of CCBs for use in chemical stabilization of trace elements in wastes and wastewater that commonly exist as oxyanions in nature, such as arsenic, boron, chromium, molybdenum, selenium, and vanadium. The specific chemical reaction responsible for the elemental stabilization is the formation of ettringite and similar hydration products which are formed on hydration of many CCBs. While chemical stabilization may be accompanied by solidification of the waste treated, solidification is not a requirement for the stabilization of the oxyanionic species as described in this standard. This standard addresses the use of CCBs as a stabilizing agent without addition of other materials; however, stabilization or chemical fixation may also be achieved by using combinations of CCBs and other products such as lime, cement kiln dust, cement, and others. In most cases, these materials would be used to improve the chemical fixation by raising the pH and providing a calcium source.

The results of the ASTM ballot were quite varied, with the majority of the comments indicating minor revisions or additions to the standard. There were comments submitted from CARRC members and EPA that reflected the concern of CARRC researchers and led to a postponement of further balloting. These comments addressed two primary issues: 1) the relationship of chemical reactions and solidification of wastes forms and 2) field proof of chemical mechanisms responsible for waste stabilization. These issues are extremely important to address in a technically appropriate manner, but required a greater effort than was economically feasible for CARRC during 1997. The following approach was developed for consideration by CARRC members and other potential partners.

- Partner with federal agencies such as EPA to identify stabilized wastes that incorporated CCBs as the primary stabilizing agent.

- Seek funding from EPA or ASTM Institute for Standards Research (ISR) to collect and evaluate these field materials.

- Gain permission from the appropriate authorities to collect samples of stabilized wastes, information on the character of the waste prestabilization, and information on the stabilization agent and process.

- Evaluate the collected stabilized waste samples for CCB hydration products and other crystalline materials, and evaluate the physical nature of the stabilized waste and mobility of constituents identified in original waste.

- Use the accumulated data to finalize the ASTM Standard Guide for “Use of Coal Combustion Byproducts for Stabilization of Wastes Containing Arsenic, Boron, Chromium, Molybdenum, Selenium, Vanadium, and Other Oxyanionic Species” and develop a CARRC topical report detailing analytical results.

4.3.2 Determination of Rate of Hydration and Reaction Products

Tasks planned for 1997 were focused on the development of analytical procedures to identify specific mineral transformations that may be associated with heat of hydration events. The first steps were to perform additional heat of hydration experiments that followed the heat of hydration events over time and the associated pH development.

The remainder of the experiments for this task were delayed until 1998 because of the unavailability of XRD instrumentation in EERC laboratories destroyed by flooding. The replacement XRD instrumentation was scheduled for installation in early 1998. Mr. Kurt Eylands was instrumental in working with laboratory staff to specify new instrumentation to facilitate CARRC analytical tasks. As a result, the instrumentation ordered will accommodate rapid scanning of samples which will allow CARRC researchers to perform the analyses required to identify mineral transformations in hydrating CCBs with relative ease.

4.3.3 Impact of Coal Fly Ash on Set Time of Concrete

The scope of this task is to look at the effects of fly ash on the set time of concrete using ASTM C403 “Time of Setting of Concrete Mixtures by Penetration Resistance.” Three fly ash sources were chosen for this task.

Data on a control mixture, the fly ash mixtures with only air entrainment, and approximately four concrete mixtures using additional chemical admixtures have been assessed. Additional work will be reported in a topical report for CARRC members and DOE.

4.3.4 Special Projects

Two primary activities were performed under the CARRC special projects in 1997.

4.3.4.1 Assessment of Fly Ash Variability

A special project was performed for Environmental Resource Corporation to evaluate the intra laboratory variability of coal fly ash from several midwestern utilities. The statistical analyses consisted of averaged values with a plus/minus standard deviation and the lowest and highest value used in the analyses. In most instances, there were three to five samples represented from each electric power plant station. All available analyses for all samples received for this special project were reported to Environmental Resource Corporation and were placed in the most recent version of CAPD, so they could accessed by CARRC members as part of the general population of the database.

The first statistical determination of the fly ash samples was the average value. The average, also referred to as arithmetic mean or just mean, is the sum of the numbers divided by the total number of numbers. The mean gives us a measure of central tendency of a list of numbers, but the mean tells nothing about the spread of the numbers in the list. In addition to a measure of central tendency, another kind of measure is used, called a measure of variation, which describes how much the numbers vary. The type of measure of variation here is the range. The range is the difference between the largest and smallest number in a series. The range has the advantage of being very easy to compute and gives a rough estimate of the variation among the data in the sample set. However, it depends only on the two extremes and tells nothing about how the other data are distributed between the extremes.

Results of the statistical evaluation will be reported on approval of the project sponsor; however, sample identity will remain confidential. It is important to note that the statistical evaluation described here can also be performed using the statistical package included in CAPD. Any CARRC member can select a data set based on any parameters desired, including names of your own sources, coal type, boiler type, or any analytical result, and perform a similar evaluation in CAPD. Results of any CAPD search, statistical evaluation, or other manipulation can be saved to disk for printing, plotting, or input to other software.

4.3.4.2 Development and Presentation of a Workshop on CCB Utilization

This workshop was developed to provide a combination of activities that would prompt the participants to gain a level of comfort with CCBs in commercial practice. The format of the workshop included a small-scale field demonstration, classroom instruction, and invited speakers.

Industrial sponsors were sought to provide funding or in-kind costs, and these contributions were matched through the CARRC DOE JSRP funding.

The agenda for the workshop was developed to meet two primary objectives: 1) to provide general “materials” information about CCBs and 2) to address issues that are of key interest to the regulatory community.

Since CCB utilization is an extremely broad topic and the subject of numerous reports, proceedings, and books, it was determined during the workshop planning phases that a reference book would be valuable to the CCB industry and specifiers and users of CCBs. A large number of technical, regulatory, promotional, and summary documents covering many aspects of CCB utilization were reviewed, and a limited number were selected based on technical merit, general applicability, and topic area. Where necessary, permission was obtained to reprint documents that were not public record. The selected documents were categorized and placed into a book titled “Coal Combustion By-Products Sourcebook.” The sourcebook was provided to each workshop participant and all CARRC members.

Workshop presenters were identified to discuss the topics included in the workshop agenda. The identified presenters included several EERC researchers, other researchers, representatives of government agencies, and representatives from industry. Each presenter was asked to prepare slides or overheads and hard copy documentation of the presentation. A second book, which contained this hard copy documentation, was also prepared and given to each attendee.

The first 1997 Coal Combustion By-Products Workshop was held March 20–21, 1997, in Colorado Springs, Colorado. The workshop had 35 attendees representing regulatory agencies, end users, utilities, other industrial ash generators, ash marketers, and entrepreneurs. The second Workshop was held May 8–9, 1997, in Minneapolis, Minnesota, where CARRC members were invited to attend as complimentary guests. There were 23 attendees at the Minneapolis workshop, with representation from the same groups as in Colorado.

4.3.5 Technology Transfer Activities

Technology transfer efforts during 1997 included the presentation or preparation of numerous formal papers. Additionally, CARRC researchers developed a brief seminar on the use of fly ash in concrete for the 1997 Forx Home Builders Show in Grand Forks, North Dakota, provided informal comments on the use of fly ash concrete in energy-efficient construction at a seminar presented by representatives of the Region 8 DOE office for groups considering large postflood building projects in Grand Forks, North Dakota, and provided informational packets on CCBs to a wide variety of government representatives, private citizens, end users of CCBs, and CCB industry representatives.

CARRC researchers participated in a variety of local, national, and international technical meetings, symposia, and conferences by presenting and publishing CCB-related papers. The paper topics and forums were selected to reach a range of audiences from the CCB industry and other groups. Twelve papers were published and/or presented by CARRC researchers during 1997.

4.4 January 1, 1998 – June 30, 1998, Tasks

Goals for 1998 were to:

- Advance predictive capabilities for and demonstrate ash behavior in utilization applications.
- Develop valid test procedures for CCBs.
- Promote beneficial reuse of CCBs through technology and information transfer to industrial, public, and governmental sectors.
- Facilitate development of reasonable CCB standards and regulations, emphasizing product performance and reasonable environmental considerations.

4.4.1 Development of Standards and Specifications

CARRC participation in activities of ASTM continued in 1998. In 1997, progress was made on standards for the SGLP and for utilization of CCBs for stabilization of wastes containing hazardous oxyanions. Both standards were revised based on input from CARRC members and other reviewers during the first part of 1998. The standards will be submitted for ballot in Fall 1998.

4.4.2 Evaluation of Role of CCBs in Waste Stabilization

During the past year, CARRC staff wrote and submitted for ballot a draft ASTM standard titled “Use of Coal Combustion Byproducts for Stabilization of Wastes Containing Arsenic, Boron, Chromium, Molybdenum, Selenium, Vanadium, and Other Oxyanionic Species.” In discussion of the comments for the first ballot, several key points were made regarding the lack of field information supporting the scientific premise used in writing the draft standard. That premise is that CCBs contribute the starting materials for ettringite formation which is responsible for the stabilization of specific elements. It is proposed to develop field information on stabilized wastes, so the premise on which the standard was based can be confirmed or refuted. It is proposed to identify several waste stabilization sites that incorporated CCBs as part of the stabilization agent, obtain samples of the stabilized waste form, and evaluate the samples for the presence of ettringite and other key CCB hydration products.

EPA representatives were contacted to assess their interest in the development standards on the use of CCBs in stabilization and in developing technical information on waste stabilization using CCBs. Mr. Ed Barth of the Cincinnati EPA Office expressed interest in this “unmet need,” and CARRC researchers continue to work with him to identify opportunities to evaluate stabilized wastes postplacement.

4.4.3 Development of a CCB Utilization Forum

An interactive forum on using CCBs in public works, organized by the EERC, was held in Salt Lake City, Utah, on April 16–17, 1998. WRAG, a regional industry group, was the lead financial sponsor for the effort, but other industrial partners also provided financial and in-kind sponsorship. The effort was performed with DOE JSRP funding through CARRC.

The goal of the forum was to provide an opportunity for interested parties in government and industry to interact and learn about CCB utilization from experts in the field of public works. This was accomplished by inviting speakers to address specific topics over a 1½-day schedule and holding a facilitated discussion period at the close of the forum.

Speakers included world-renowned experts in a variety of areas related to the use of CCBs in concrete and soil applications. A Plenary Session included Bryant Mather, U.S. Army Corps of Engineers, Barry Stewart, ACAA, William Aljoe, DOE Federal Energy Technology Center (FETC), and David Hassett, EERC. Other speakers included representatives of several different state agencies, regional universities, and regional engineers and contractors. Debra Pflughoeft-Hassett, EERC, facilitated the closing discussion session.

The forum attracted approximately 50 participants from the utility and CCB marketing industries and associated industries. Several participants commented on the excellent quality of the speakers and presentations. The WRAG members' critique of the forum was also very positive during its membership meeting following the forum. WRAG members voted to work with the EERC in a similar effort related to CCB utilization in mining, tentatively scheduled for April 1999.

4.4.4 Assessing the Use of Bottom Ash in Road Base Construction

CARRC staff worked with local CCB marketers and engineers to develop a test plan to evaluate the performance of bottom ash in the construction of road base in North Dakota. On approval, this work will begin in fall 1998.

Other CARRC tasks are continuing in 1998–1999. New members are anticipated to join CARRC in 1999. An annual meeting will be held in October 1998 to develop the work plan for 1999.